# International Standard

**ISO 7258**

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# Polytetrafluoroethylene (PTFE) tubing for aerospace applications — Methods for the determination of the density and relative density

*Tubes en polytétrafluoréthylène (PTFE) à usage aéronautique — Méthodes de détermination de la masse volumique et de la densité*

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# Foreword

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It has been approved by the member bodies of the following countries :

| | | |
|---|---|---|
| Australia | Czechoslovakia | Romania |
| Austria | Egypt, Arab Rep. of | South Africa, Rep. of |
| Belgium | France | Spain |
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| Canada | Italy | United Kingdom |
| China | Netherlands | USA |

No member body expressed disapproval of the document.

# Polytetrafluoroethylene (PTFE) tubing for aerospace applications — Methods for the determination of the density and relative density

## 0 Introduction

Density and relative density are frequently used to follow the variations in the physical structure of specimens and in calculations of the amount of material necessary to fill a given volume. Density is the preferred property relating the mass and volume of an object, specimen or material. These properties may also be useful in determining uniformity among samples or specimens.

## 1 Scope and field of application

This International Standard specifies three methods for the determination of the density and relative density of polytetrafluoroethylene (PTFE) tubing used in the manufacture of medium and high pressure, high temperature, PTFE hose assemblies for aerospace applications :

Method A — Density gradient column method without a preconditioning heating and cooling cycle.

Method B — Displacement of water and determination of change of weight method.

Method C — Density gradient column method with a preconditioning heating and cooling cycle.

NOTE — The term "Apparent Specific Gravity" is used within the PTFE hose industry for the density or relative density determined by methods A and B. The term "Relative Specific Gravity" is similarly used for the results of method C.

## 2 Definitions

**2.1 density** : The mass per unit volume of material at $t$ °C density is expressed as follows :

density, $t$ °C : $\varrho_t$ in grams per cubic centimetre or grams per millilitre

For the purpose of this International Standard, $t$ is equal to 25 °C.

**2.2 relative density** : The ratio of the mass in air of a given volume of material to the mass in air of an equal volume of a reference material at the same temperature; it is expressed as

relative density, $t/t$ °C : $d_t^t$

where $t$ is the temperature in degrees Celsius. For the purpose of this International Standard, $t$ is equal to 25 °C.

NOTES

1 Throughout this International Standard, the term "relative density" should be taken as meaning relative density with respect to water. In this context, the English term "specific gravity" is often used for "relative density" when the reference material (see 2.2) is water.

2 Density in grams per cubic centimetre may be converted to relative density with respect to water as follows :

$$d = \frac{\varrho_t}{\varrho_t'}$$

where

$d$ is the relative density;

$\varrho_t$ is the density of the specimen;

$\varrho_t'$ is the density of water, at temperature $t$.

At a temperature of 25 °C, $\varrho_t' = 0{,}997\,1$ g/cm³.

## 3 Apparatus

### 3.1 Methods A and C

**3.1.1 Density gradient tube**, a suitable graduate with stopper, preferably ground glass.

**3.1.2 Constant temperature bath**, a means of controlling the temperature of the liquid in the tube at 25 ± 1 °C. A thermostatted water jacket around the tube is a satisfactory and convenient method of achieving this.

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**3.1.3 Glass floats,** a number of calibrated glass floats covering the density range to be studied and approximately evenly distributed throughout this range.

**3.1.4 Pycnometer,** used in determining the densities of the standard floats.

**3.1.5 Hydrometers,** a set of suitable hydrometers covering the range of densities to be measured. These hydrometers should have 0,001 density graduations.

**3.1.6 Analytical balance,** with a sensitivity of 0,1 mg.

**3.1.7 Siphon or pipette arrangement,** for filling the gradient tube. This piece of equipment should be constructed so that the rate of flow of liquid may be regulated to 10 ± 5 ml/min.

## 3.2 Method B

**3.2.1 Analytical balance,** a balance with a precision within 0,1 mg, accuracy within 0,05 % relative (that is, 0,05 % of the weight of the specimen in air), and equipped with a stationary support for the immersion vessel above the balance pan ("pan straddle").

**3.2.2 Wire,** a corrosion-resistant wire for suspending the specimen.

**3.2.3 Immersion vessel :** A beaker or other wide-mouthed vessel for holding the water and immersed specimen.

## 4 Immersion liquids

The liquid or solution with which the specimen comes into contact during the measurement should have no solvent or chemical effect on the specimen and should not be absorbed by the specimen in any significant quantity.

## 4.1 Methods A and C

Mixtures of two liquids selected from those listed in the table, suitable for the preparation of the required density gradient.

Table — Liquid systems for density gradient columns

| Liquid system | Density range g/ml |
|---|---|
| Tetrachloroethylene/tribromomethane | 1,62 to 2,89 |
| Bromobenzene/tribromomethane | 1,50 to 2,89 |
| Chlorobenzene/tribromomethane | 1,11 to 2,89 |

## 4.2 Method B

Substantially air-free distilled or demineralized water, containing two drops of wetting agent.

NOTE — Water may be rendered substantially air-free by boiling and cooling or by shaking under vacuum in a heavy-walled vacuum flask.

## 5 Specimens

## 5.1 Methods A and C

The sample shall be a representative cross-section of the size, thickness and construction of the tubing under test. The specimens of manufactured polytetrafluoroethylene resin should be quite small, a perimeter of 9,5 mm to 16 mm being adequate. In order to differentiate between specimens, they may be cut from the sample in a variety of simple geometric shapes. Holes or cracks in the specimens will entrap air and cause bubbles. This will influence the relative density and should be avoided. Moreover, the specimens should be cut with a sharp knife or razor to prevent ragged edges.

## 5.2 Method B

The test specimen shall be a single piece of the material under test, of any size and shape that can conveniently be prepared and tested, provided that its volume shall be not less than 1 cm³ and its surface and edges shall be made smooth. The thickness of the specimen should be at least 1 mm for each 1 g of mass. Usually a specimen weighing 1 to 5 g will be found convenient, but specimens up to approximately 50 g may be used. Care should be taken in cutting specimens to avoid changes in density resulting from compressive stresses or frictional heating.

## 6 Procedures

## 6.1 Method A

### 6.1.1 Preparation of standard glass floats

Prepare glass floats (3.1.3) by any convenient method such that they are fully annealed, approximately spherical, have a maximum diameter less than one quarter the inside diameter of the column and do not interfere with the test specimens. Prepare a solution (400 to 600 ml) of the liquids (4.1) to be used in the gradient tube (3.1.1) such that the density of the solution is approximately equal to the desired lowest density. When the floats are at room temperature, drop them gently into the solution. Save the floats that sink very slowly, and discard those that sink very fast or save them for another tube. If necessary to obtain a suitable range of floats, grind selected floats to the desired density by rubbing the head part of the float on a glass plate on which is spread a thin slurry of silicon carbide of size 38 μm (400 mesh) maximum or other appropriate abrasive. Progress may be followed by dropping the float in the test solutions at intervals and noting its change in rate of sinking.

### 6.1.2 Calibration of standard glass floats

**6.1.2.1** Place a tall cylinder in the constant temperature bath (3.1.2) maintained at 25 ± 1 °C. Then fill the cylinder about two thirds full with a solution of two suitable liquids, the density of which can be varied over the desired range by the addition of either liquid to the mixture. After the cylinder and solution have attained temperature equilibrium, place the float in the solution, and if it sinks, add the denser liquid by suitable means with good stirring until the float reverses direction of movement. If the float rises, add the less dense liquid by

suitable means with good stirring until the float reverses direction of movement.

**6.1.2.2** When reversal of movement has been observed, reduce the amount of the liquid additions to that equivalent to 0,000 1 g/cm³ density. When an addition equivalent to 0,000 1 g/cm³ density causes a reversal of movement, or when the float remains completely stationary for at least 15 min the float and liquid are in satisfactory balance. The cylinder shall be covered whenever it is being observed for balance, and the liquid surface shall be below the surface of the liquid in the constant temperature bath. After vigorous stirring, the liquid may continue to move for a considerable length of time; make sure that the observed movement of the float is not due to liquid motion by waiting at least 15 min after stirring has stopped before observing the float.

**6.1.2.3** When balance has been obtained, fill a freshly cleaned and dried pycnometer (3.1.4) with the solution and place it in the 25 ± 1 ° C bath for sufficient time to allow temperature equilibrium of the glass. Determine the density of the solution by normal methods and make *in vacuo* corrections for all weighings. Record this as the density of the float. Repeat the procedure for each float.

### 6.1.3 Gradient tube preparation (stepwise addition method)

**6.1.3.1** Using the two liquids (see 4.1) that will give the desired density range and sensitivity ($S$) in grams per cubic centimetre per millimetre, prepare four or more solutions such that each differs from the next heavier by $80S$ g/cm³. The number of solutions will depend upon the desired density range of the column and shall be determined as follows :

Number of solutions to prepare density-gradient column :

$$N = \frac{(1 + \varrho_2 - \varrho_1)}{80S}$$

where

$\varrho_1$ is the lower limit of density range desired;

$\varrho_2$ is the upper limit of density range desired, and

$S$ is the sensitivity, in grams per cubic centimetre per millimetre.

NOTE — Round off the value of $(1 + \varrho_2 - \varrho_1)/80S$ to the nearest whole number.

To prepare these solutions, proceed as follows :

Using the hydrometers (3.1.5), mix the two liquids in the proportions necessary to obtain the desired solutions. Remove the dissolved air from the solutions by gentle heating or an applied vacuum. Then check the density of the solutions at 25 ± 1 °C by means of the hydrometers and, if necessary, add the appropriate air-free liquid until the desired density is obtained.

NOTE — In order to obtain a linear gradient in the tube, it is very important that the solutions be homogeneous and at the same temperature

when their densities are determined. It is also important that the density difference between the solutions consecutively introduced into the tube be equal.

**6.1.3.2** By means of a siphon or pipette (3.1.7), fill the gradient tube with an equal volume of each liquid starting with the heaviest, taking appropriate measures to prevent air from being dissolved in the liquid. After the addition of the heaviest liquid, very carefully and slowly pour an equal volume of the second heaviest liquid down the side of the column by holding the siphon or pipette against the side of the tube at a slight angle. Avoid excess agitation and turbulence. In this manner, the "building" of the tube shall be completed.

NOTE — Density gradients may also be prepared by reversing the procedure described in 6.1.3.1 and 6.1.3.2. When this procedure is used, the lightest solution is placed in the tube and the next lightest solution is very carefully and slowly "placed" in the bottom of the tube by means of a pipette or siphon which just touches the bottom of the tube. In this manner, "building" of the tube shall be completed.

**6.1.3.3** If the tube is not already in a constant temperature bath, transfer the tube, with as little agitation as possible, to the constant temperature bath maintained at 25 ± 1 °C. The bath level should approximately equal that of the solution in the tube, and provision should be made for vibrationless mounting of the tube.

**6.1.3.4** For every 250 mm of length of tube, dip a minimum of five clean calibrated floats, spanning the effective range of the column, into the less dense solvent used in the preparation of the gradient tube and add them to the tube. By means of a stirrer (for example, a small coiled wire or other appropriate stirring device) mix the different layers of the tube gently by stirring horizontally until the least dense and most dense floats span the required range of the gradient tube. If at this time it is observed that the floats are "bunched" together and not spread out evenly in the tube, discard the solution and repeat the procedure. Then cap the tube and keep it in the constant temperature bath for a minimum of 24 h.

**6.1.3.5** At the end of this time, plot the density of floats against the height of floats to observe whether or not a fairly smooth and linear curve is obtained. Some small irregularities may be seen, but they should be slight. Whenever an irregular curve is obtained, the solution in the tube shall be discarded and a new gradient prepared.

NOTE — Gradient systems may remain stable for several months. A daily check of the original calibration will reveal when instability has been reached.

### 6.1.4 Measurement of density

**6.1.4.1** Wet three representative test specimens with the less dense of the two liquids used in the tube and gently place them in the tube. Allow the tube and specimens to reach equilibrium, which may require 10 min or more.

**6.1.4.2** When a graduated tube is used, read the height of the floats and specimens by using a line through their centre of volume. When a cathetometer is used, measure the height of the floats and specimens from an arbitrary level using a line

through their centre of volume. If equilibrium is not obtained, the specimen may be imbibing the liquid.

**6.1.4.3** Old specimens can be removed without destroying the gradient by slowly withdrawing a wire screen basket attached to a long wire. This can be conveniently done by means of a clock motor. Withdraw the basket from the bottom of the tube and, after cleaning, return it to the bottom of the tube. It is essential that this procedure be performed at a slow enough rate (approximately 30 min/300 mm length of column) so as not to disturb the density gradient.

### 6.1.5 Calculations

The densities of the specimens may be determined graphically or by calculation from the levels to which they settle by either of the following methods :

#### 6.1.5.1 Graphical calculation

Plot float position versus float density on a chart large enough to be read accurately to ± 1 mm and the desired precision of density. Plot the positions of the unknown specimens on the chart and read their corresponding densities.

#### 6.1.5.2 Numerical calculation

Calculate the density by interpolation as follows :

Density at height $h = \varrho_A + [(h - h_A)(\varrho_B - \varrho_A)/(h_B - h_A)]$

where

$\varrho_A$ and $\varrho_B$ are the densities of the two standard floats;

$h_A$ and $h_B$ are the distances of the two standards A and B respectively, bracketing the unknown measured from an arbitrary level;

$h$ is the distance of unknown above the same arbitrary level.

#### 6.1.5.3 Conversion to relative density

When required, the relative density shall be calculated from the density value obtained in 6.1.5.1 or 6.1.5.2 by using the formula given in note 2 of 2.2.

## 6.2 Method B

### 6.2.1 Conditioning

The test specimens shall be conditioned at 25 ± 1 °C and 50 ± 5 % relative humidity for not less than 1 h prior to test.

### 6.2.2 Measurement of relative density

**6.2.2.1** Using the analytical balance (3.1.6), weigh the specimen in air to the nearest 0,1 mg or 0,5 % relative, whichever is greater.

**6.2.2.2** Attach to the balance a piece of fine wire (3.2.2) of sufficient length to reach from the hook above the pan to the support for the immersion vessel (3.2.3). Attach the specimen to the wire such that it is suspended about 25 mm above the vessel support.

NOTE — The specimen may be weighed in air after hanging from the wire. In this case, record the weight of the specimen $W_0$ = (weight of specimen + wire, in air) — (weight of wire in air).

**6.2.2.3** Mount the immersion vessel on the support, and completely immerse the suspended specimen in water (see 4.2) at a temperature of 25 ± 1 °C. The vessel shall not touch the wire or specimen. Remove any bubbles adhering to the specimen or wire, paying particular attention to holes in the specimen. Usually, these bubbles can be removed by rubbing them with another wire. If the bubbles cannot be removed by this method or if bubbles are formed continuously (as from dissolved gases), the use of vacuum is recommended (see the note). Weigh the suspended specimen to the required precision (see 6.2.2.1). (It may be necessary to change the sensitivity adjustment of the balance to overcome the damping effect of the immersed specimen.) Record this weight as $W_1$ (the weight of the specimen and the partially immersed wire in liquid).

NOTE — Some specimens may contain absorbed or dissolved gases, or irregularities which tend to trap air bubbles; any of these may affect the density values obtained. In such cases, the immersed specimen may be subjected to vacuum in a separate vessel until evolution of bubbles has substantially ceased before weighing. It also has to be demonstrated that the use of this technique leads to results of the required degree of precision.

**6.2.2.4** Weigh the wire in water with immersion to the same depth as used in the previous step. It is convenient to mark the level of immersion by means of a shallow notch filed in the wire. The finer the wire, the greater the tolerance which may be permitted in adjusting the level of immersion between weighings. With wire SWG No. 40 or finer, disregard its degrees of immersion and use the apparent weight of the wire in air as $W$. If the wire is left attached to the balance arm during a series of determinations, the apparent weight $W_0$ may be determined with the aid of a tare on the other arm of the balance. In such cases, care shall be taken that the change of weight of the wire (for example, from visible water) between readings does not exceed the desired precision. Record this apparent weight as $W$ (weight of the wire in liquid).

### 6.2.3 Calculation

Calculate the relative density of the plastic as follows :

$$d\,_{25\ ^{\circ}C}^{25\ ^{\circ}C} = \frac{W_0}{W_0 + W - W_1}$$

where

$W_0$ is the apparent weight of specimen, without wire, in air;

$W$ is the apparent weight of partially immersed wire;

$W_1$ is the apparent weight of specimen completely immersed and of the wire partially immersed in liquid.